



ifw
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of

Craig Stuart Valentine, et al.

Serial No.: ~~10/670,568~~ 10760568

Filed: January 20, 2004

Title: STABILIZED POLYESTER COMPOSITIONS AND) Examiner:
MONOFILAMENTS THEREOF FOR USE IN A PAPER)
MACHINE CLOTHING AND OTHER INDUSTRIAL)
FABRICS)

Group:1714

CLAIM FOR PRIORITY

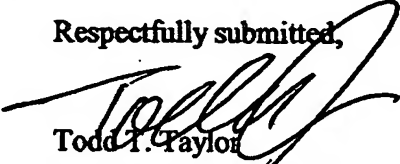
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

Applicants hereby claim the priority of Great Britain Patent Application Serial No.
0117830.0, filed July 21, 2001, under the provisions of 35 U.S.C. 119.

A certified copy of the priority document is enclosed herewith.

Respectfully submitted,


Todd T. Taylor
Registration No. 36,945
Attorney for Applicant

TTT/ap

TAYLOR & AUST, P.C.
142 S. Main St.
P.O. Box 560
Avilla, IN 46710
Telephone: 260-897-3400
Facsimile: 260-897-9300

Encs.: Priority Document
Return postcard

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States
Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, P.O.
Box 1450, Alexandria, VA 22313-1450, on: October 13, 2005.

Todd T. Taylor, Reg. No. 36,945
Name of Registered Representative


Signature

October 13, 2005
Date

BEST AVAILABLE COPY

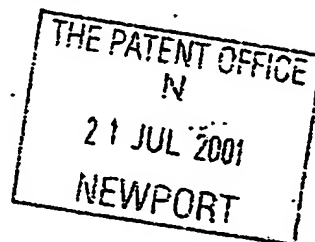
**CERTIFIED COPY OF
PRIORITY DOCUMENT**

21 JUL 2001

VUE

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road
Newport

Gwent NP9 1RH

1. Your reference

MG/NH/P/19225.GB

2. Patent application number

(The Patent Office will fill in this part)

0117830.0**3. Full name, address and postcode of the or of each applicant (underline all surnames)**

Voith Fabrics Heidenheim GmbH & Co. KG,
Kurze Strasse 11,
89522 Heidenheim,
Germany.

Patents ADP number (if you know it)

If the applicant is a corporate body, give the
country/state of its incorporation

7841125001

23JUL01 E647096-8 D00350
P01/7700 0.00-0117830.0

Heidenheim, Germany

4. Title of the invention

Stabilised Polyester Compositions and Monofilaments
thereof for use in Papermachine Clothing and other
Industrial Fabrics

5. Name of your agent (if you have one)

WILSON GUNN M'CAW

"Address for service" in the United Kingdom
to which all correspondence should be sent
(including the postcode)

WILSON GUNN M'CAW
41-51 ROYAL EXCHANGE,
CROSS STREET,
MANCHESTER.
M2 7BD.

Patents ADP number (if you know it)

7153927001 ✓

If you are declaring priority from one or more
earlier patent applications, give the country
and the date of filing of the or of each of these
earlier applications and (if you know it) the or
each application number

Country

Priority application number
(if you know it)Date of filing
(day / month / year)

If this application is divided or otherwise
derived from an earlier UK application,
give the number and the filing date of
the earlier application

Number of earlier application

Date of filing
(day / month / year)

Is a statement of inventorship and of right
to grant of a patent required in support of
this request? (Answer 'Yes' if

YES

a) any applicant named in part 3 is not an inventor, or

b) there is an inventor who is not named as an
applicant, or

c) any named applicant is a corporate body.

See note (d))

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

BEST AVAILABLE COPY

Continuation sheets of this form

Description

21✓

Claim(s)

Abstract

Drawing(s)

02+2✓

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11.

I/We request the grant of a patent on the basis of this application.

Signature

Date

20/07/01

Mr. M. Goodwin

12. Name and daytime telephone number of person to contact in the United Kingdom

Mr. M. Goodwin
0161-827-9400

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.

Stabilised Polyester Compositions and Monofilaments thereof for use in
Papermachine clothing and other Industrial fabrics

The present invention relates to stabilised polyester compositions and
5 monofilaments thereof for use in papermachine clothing and other industrial
fabrics.

Monofilaments manufactured from polyethylene terephthalate (or PET)
are used extensively in the production of papermachine clothing and other
industrial fabrics. A paper machine typically comprises three sections. In
10 the forming section, where the cellulosic fibers are presented to a forming
fabric in the form of a slurry, the fabrics are predominantly constructed from
polyester monofilaments, more specifically PET. In the forming section of
the papermachine the temperature rarely exceeds 60°C and the fabric is
subjected to severe wear from suction boxes used to withdraw water from
15 the paper web, such that the fabric life is seldom over 120 days. The paper
sheet is transferred from the forming section into the press section of the
papermachine and at this point the solids content of the slurry is
approximately 20%. Here, the paper sheet passes through a series of nip
rolls or shoe presses, and due to the need for resilience, polyamides have
20 been the material of choice. The paper sheet has about 40% solids content
as it is transferred from the press section into the dryer section of the
papermachine.

In the dryer section, a textile fabric holds the paper sheet against steam-heated cylinders. The temperature of the cylinder surface can exceed 120°C and the evaporation of water from the sheet ensures that the relative humidity remains at 100%. Fabrics composed of PET are conventionally used for most dryer fabric applications. However, towards the end of the paper machine, as the solids content approaches 80 to 90%, the cooling effect of the evaporation is reduced and the temperature to which the fabric is actually exposed increases such that the PET fabric is now subjected to significant degradation. In most applications, the life of a typical PET fabric can be in excess of 12 months. However, under these extreme conditions service life is reduced significantly.

In order to extend the service life of dryer fabrics exposed to these conditions, suppliers to the industry have used an array of materials as the constituent material of the dryer fabric. Polyphenylene sulphide (or PPS) provides excellent thermal, hydrolytic and oxidative stability. However, the PPS polymer is significantly more expensive than PET. Monofilament extrusion of PPS is more problematic, leading to a higher percentage of product rejections and therefore higher production costs.

Copolyesters derived from 1,4-cyclohexane dimethanol, terephthalic acid, isophthalic acid and esters thereof, have been suggested as a cheaper alternative. U.S. Patent No. 5,169,499 teaches the use of such copolyesters to improve the hydrolytic stability of papermachine clothing.

The large cyclohexane moiety present in the polymer backbone serves to provide steric hindrance to the hydrolytic cleavage of the ester bond.

However, the cyclohexane ring also serves to increase the susceptibility of such polyesters to oxidative degradation. It is generally accepted that the

oxidation of polymers follows a free radical chain reaction mechanism that

is initiated by abstraction of a hydrogen atom from the polymer, forming an alkyl radical. This alkyl radical can very quickly react with oxygen to form

an alkyl peroxy radical, which propagates additional reactions. Each

cyclohexane ring, whilst providing steric hindrance, also introduces two

tertiary hydrogen atoms into the backbone of the polymer; that is two hydrogen atoms that are each bonded to a tertiary carbon. Due to the

effects of electron withdrawal, the carbon hydrogen bond strength is reduced, such that the abstraction of these hydrogen atoms is much more

likely at elevated temperatures. Hence, polyesters that contain this type of

cyclohexane moiety are more prone to oxidative degradation than PET which has no tertiary hydrogen atoms in its polymer backbone.

The art of stabilizing polymers to oxidation at elevated temperatures is extensive. U.S. Patent No. 5,763,512 teaches the use of a combination

of a sterically hindered phenol and a specific organic phosphite for the stabilization of polyamides, polyesters or polyketones against oxidative,

thermal and/or light induced degradation. Sterically hindered phenols and

other organic compounds that can form resonance-stabilized radicals, are

known to scavenge alkyl and alkyl peroxy radicals formed during the oxidation of a polymer, and are commonly termed primary anti-oxidants. Tri-aryl phosphites and thioester compounds react with hydroperoxide moieties formed during oxidation, and are commonly referred to as secondary anti-oxidants. Polymer Science and Engineering, Vol. 30, No. 17, page 1041 by A. Aurebach et al cited herein, describes blends of PCT and the use of certain anti-oxidants to improve melt stability.

U.S. Patent No. 5,981,062 attempts to improve the stability of such polyesters through blending with polyamides, more specifically the blending of polyesters based upon a polyhydric alcohol of 1,4-cyclohexane dimethanol with 5 to 20% of a polyamide, preferably PA66. The blends are shown to improve the oxidative and hydrolytic stability of monofilaments manufactured therefrom.

Polyamide 66 is known to form gels if held at elevated temperatures for an extended period of time (see Nylon handbook published by Hanser/Gardner Publications 1995, Chapter 3, Page 55). Polyesters derived from 1,4-cyclohexane dimethanol and terephthalic acid (or its esters); i.e. PCT, or polyesters derived from 1,4-cyclohexane dimethanol and terephthalic and isophthalic acid (or their esters); i.e. PCTA, have melting points of 295°C and 285°C respectively. These high melting points necessitate high temperature processing and melt temperatures which can be in excess of 300°C. We have seen that this will lead to some degree of gel formation

with polyamide 66 and can result in some thermal degradation of PA6. Gels occur during melt processing when cross-links form between individual polymer chains. In monofilament extrusion, the presence of gels leads to diameter variation at localized sections of the filaments that are very brittle and exhibit poor mechanical properties. It is known that these filaments will break under the normal loads experienced in a weaving process. Such filaments are of an unacceptable quality.

It is known to those skilled in the art that polyesters and polyamides are generally incompatible, and will tend to exhibit phase separation in the melt. Phase separation would induce micro-voids and various structural defects, the effect of which is to introduce weak points observed as tenacity variation along the length of the monofilament. These defects can also affect the drawing process, reducing the efficiency of monofilament manufacture.

In addition, the blends alone do not provide sufficient oxidative stability to match that of the industry standard polyester, PET, ensuring that they, and the monofilaments and textile structures derived from them, cannot be used universally. Hence, there remains a need for further stabilization of such polyesters, as described herein against thermo-oxidative degradation.

Textile structures formed from such monofilaments may be woven for a plurality of said filaments, or they may be constructed from helical spiral

coils of the monofilaments linked together by pintle yarns, a process that is described in U.S. Patent No. 4,423,543.

It is an objective of the present invention to provide a PCTA copolymer for use in papermachine clothing and/or other industrial fabrics, having good dry heat and hydrolysis resistance.

According to a first aspect of the present invention there is provided a polyester composition comprising from 90 to 97% by weight of at least one polyester derived from the condensation of 1,4-cyclohexane dimethanol and at least one dicarboxylic acid or an ester thereof, from 0.1 to 5% of at least one primary anti-oxidant defined as an alkyl and/or alkyl peroxy radical scavenger, from 0.1 to 5% of at least one secondary anti-oxidant defined as a compound capable of decomposing a hydroperoxide, from 1 to 4% of at least one polyamide terpolymer.

The composition further preferably comprises from 0.5 to 2% of at least one hydrolysis stabilizer.

According to a second aspect of the present invention there is provided an article of papermachine clothing comprising the polyester composition of the aforesaid first aspect of the invention.

According to a third aspect of the present invention there is provided a monofilament comprising the polyester composition of the first aspect of the invention.

The polymer compositions of the invention exhibit comparable

resistance to thermo-oxidative degradation to standard PET and vastly superior hydrolytic degradation, particularly at elevated temperatures, and improved property uniformity. Consequently the polymer compositions of the invention may be processed using standard equipment with consistent properties.

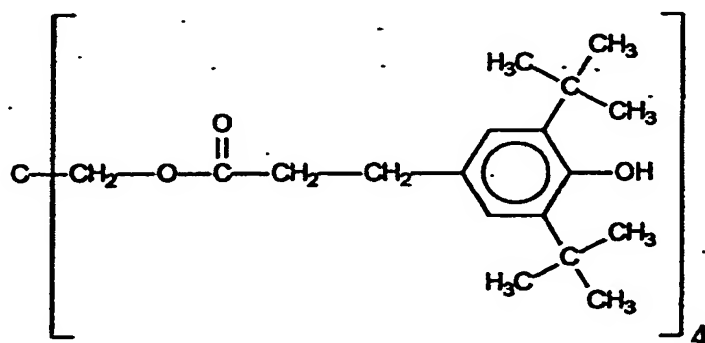
The polyester compositions (which term as used herein includes copolyesters) of the present invention are those containing a cyclohexane moiety in the polymer backbone. The polyester preferably comprises the condensation product of 1,4-cyclohexane dimethanol and terephthalic acid and/or an ester derivative of said acid. Ideally the polyester comprises the condensation product of 1,4-cyclohexane dimethanol terephthalic and isophthalic acids and/or their ester derivatives. Suitable commercially available polyesters are those available from Eastman Chemical Co., of Kingsport Tennessee under the trade marks THERMX 3879 for PCT and THERMX 13319 for PCTA respectively.

The primary anti-oxidant may comprise from 0.1 to 5% by weight of the blend, and the secondary anti-oxidant may comprise from 0.1 to 5% by weight of the blend. The term "primary anti-oxidant" refers to a material that by way of its chemical composition can readily react with alkyl peroxy radicals forming more stable radicals that do not further propagate the chain reaction. These radicals can undergo further reaction with additional radicals to prevent them from propagating the oxidation chain. The most efficient

primary anti-oxidants, by way of this reaction, are regenerated. The term "secondary anti-oxidant" refers to a class of additives that by way of their chemical composition can react with hydro-peroxide moieties formed as a result of the oxidation of a polymer, thus neutralizing these highly reactive species. Details of mechanisms involved in preventing oxidation are included in "Plastics Additives, Chapter 1 published by Hanser (1993)".

The primary anti-oxidant is preferably a hindered phenolic compound.

The type of primary anti-oxidant described in this art is exemplified by, but not limited to the following:

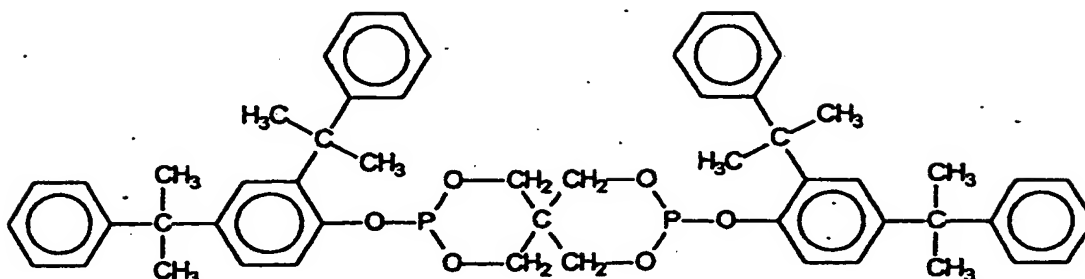


The phenolic anti-oxidant illustrated above, i.e. pentaerythrityl (tetrakis-3-(3,5-di-tert.-butyl-4-hydroxy phenyl) propionate), CAS Number 6683-19-8, is sold under the trade name IRGANOX 1010 by Ciba Corporation.

The secondary anti-oxidant is preferably a phosphite.

The type of secondary anti-oxidant described in this art is exemplified

by, but not limited to the following:



The phosphite secondary anti-oxidant illustrated above; i.e. Bis (2,4-dicumylphenyl) pentaerythritol diphosphite, CAS No. 154862-43-8, is sold under the trade name DOVERPHOS® S-9228 by Dover Chemicals, Dover, Ohio.

The hydrolysis stabilizer added to the blend was chosen for its ability to neutralize the carboxyl end groups of the polyester and is added in quantities to minimise the problems associated with manufacturing monofilaments from the blend. The composition may comprise said hydrolysis stabiliser in an amount from 0.5 to 2% by weight. The preferred stabilizer is exemplified by, but not limited to, the class of compounds known as carbodiimides. These compounds may be used in the monomeric or polymeric forms. A specific example of this type of stabilizer is 2,6 diisopropylphenyl carbodiimide which is supplied under the trade name of STABAXOL I by Rhein Chemie GmbH.

The polyamide terpolymer stabilizer ideally has a melting point in the range from 120°C to 220°C. The terpolymer is added such that it

comprises from 1 to 4% by weight of the blend. An example of such a terpolymer is sold commercially by Du Pont de Nemours under the trade name ELVAMIDE 8063. The term "terpolymer" as used herein refers to a polymer composed from more than two distinct repeat units as opposed to a homopolymer with one, and a copolymer with two. For example, a polyamide terpolymer may be composed of three or more repeat units such as 6, 6,6, 11 and 12. The type and ratio of the components has a significant influence upon the properties of the material, such that they are useful. The term "polyamide" refers to any of the known polyamides, which through polymerization, can be formed into terpolymers. Examples include, but are not limited to polyamide 6, polyamide 11, polyamide 12, polyamide 6,6, polyamide 6,9, polyamide 6,10, polyamide 6,12.

The blend of a polyamide terpolymer, a primary anti-oxidant and a secondary anti-oxidant as defined herein combine synergistically to provide a significant improvement in the ability of the polyesters described to withstand oxidation at elevated temperatures. Furthermore the additives in the composition of the present invention have high compatibility to the main resin, unlike many prior art compositions, resulting in the compositions of the invention having uniform properties including colour, tensile properties and resistance to degradation.

The polyester composition blends of the invention provide improved melt extrusion and consequently the consistency of monofilaments produced

by such extrusion is improved.

An extrusion processing aid in the form of a lubricant, such as silicone, may be added to the blend, and can comprise between 0.1 and 1% by weight of the final composition.

5 The polyester compositions of the present invention are particularly useful as papermachine clothing or other industrial fabrics where the textile is likely to be exposed to elevated temperatures in the presence of water or otherwise high levels of humidity are anticipated. Such fabrics may be woven from a plurality of filaments or formed from many spiral coils linked
10 together in a construction commonly termed a spiral fabric and described in U.S. Patent No. 4,423,543.

In order that the present invention may be more readily understood specific examples thereof are set out below by means of illustration only.

15 The hydrolysis and oven ageing resistance of a composition in accordance with the invention were compared to that of a stabilized PET formulation, representing the industrial standard. Also, the hydrolysis and oven ageing performance of the PCTA formulation reported in US Patent No. 5,981,062 was included for comparison.

20 Fig.1 is a graph of tensile strength retention as a function of time, illustrating the hydrolysis resistance of one example of the invention compared with PET and PCTA. The hydrolysis test was performed as outlined in example 2; and

Fig.2 is a graph of tensile strength retention as a function of time illustrating, the thermo-oxidative stability of one example of the invention compared with PET and PCTA. The oven-aging test was performed as outlined in Example 2.

5 **Example 1**

10 In the following example, a comparison of the stabilizers that can be used is described, and their effect on the oxidative degradation of the monofilament outlined. The components in each sample were pre-blended, and then dried at 170°F for 15 hours. Monofilament extrusion was carried out on a 1" single screw extruder with an L/D ratio of 25:1. The resins were gravity fed into the extruder from a hopper, into which a positive pressure of nitrogen gas was maintained to prevent moisture ingress. The composition of the samples is provided in Table 1, and the extrusion conditions employed in this example are provided in Table 2.

15 The physical properties of this monofilament as produced were measured according to ASTM D2256-97.

20 The shrinkage was tested according to ASTM D204 with the temperature modified to 204°C. The physical properties of the samples are outlined in Table 3. Oven aging tests were carried out using a forced air oven maintained at 204°C (400°F). Lengths of the monofilaments samples were wrapped into coils approximately 5cm in diameter. The coils were tied into bundles and placed in the oven, samples being removed at set time

intervals. The physical property retention was measured as a function of tenacity on an Instron Tensile Tester. The tenacity retention data is provided in Table 3.

Table 1: Composition of various samples, including samples 1-6 manufactured following the procedure outlined in Example 1.

Sample	Control	1	2	3	4	5	6	PET
% PCTA	99.7	98.7	98.7	89.7		89.7	94.7	98.8
% Irganox 1010(hindered phenolic)		0.5					0.5	
% Nylostab S-EED (hindered amine)			0.5					
% Doverphos S-9228 (phosphite)		0.5	0.5				0.5	
% tri-phenyl phosphate								
% Polyamide 6				10				
% Polyamide 6,6					10			
% Polyamide Terpolymer						10	4	
% MB50-004 UHMW Silicone	0.3	0.3	0.3	0.3	0.3	0.3	0.3	
Staboxol I								1.2

Table 2: Extrusion and Draw conditions employed in the manufacturing of the samples 1-6.

Sample	Control	1	2	3	4	5	6
<u>Extruder Temperature Profile (C)</u>							
Throat	<60	<60	<60	<60	<60	<60	<60
Feed Zone	280	280	280	280	280	280	280
Zone 2	285	285	285	285	285	285	285
Zone 3	290	290	290	290	290	290	290
Zone 4	290	290	290	290	290	290	290
Zone 5	285	285	285	285	285	285	285
Zone 6	275	275	275	275	275	275	275
Adaptor	285	285	285	285	285	285	285
Die	275	275	275	275	275	275	275
<u>Extrusion Conditions</u>							
Extruder Pressure (psi)	1000	1000	1000	1000	1000	1000	1000
Die Pressure (psi)	720	530	635	1030	750	600	680
Torque (M.g.)	2500	2500	2000	2500	2500	1900	2200
Screw Speed (rpm)	16	17	17	16	17	17	17
<u>Drawing Conditions</u>							
Total Draw Ratio	3.33	3.33	3.33	3.33	3.33	3.33	3.33
Draw Oven Temperatures (F)							
Oven 1	300	300	300	300	300	300	300
Oven 2	380	380	380	380	380	380	380
Oven 3	430	430	430	430	430	430	430

Table 3: Physical property results for the manufactured samples 1-6.

Sample	Control	1	2	3	4	5	6	PET
Denier	3450	3492	3534	3326	3492	3462	3432	2486
Elongation @ Break (%)	33	30	34	30	26	30	30	35
Tenacity (g/d)								
Initial	2.24	2.22	2.2	2.18	2.21	1.83	1.83	3.8
24hrs	Failed	2.22	1.72	1.36	1.43	1.57		
39hrs		2.22	1.03	1.34	1.43	1.18		
45hrs		2.22	1.03	0.96	1.36	1.3		
65hrs		2.08	Failed	Failed	Failed	1.05	1.83	2.5
89hrs		1.56				1.05	1.72	2.51
113 hrs		Failed				Failed	1.45	2.52

The control referred to in Tables 1 to 3 is PCTA as described in US 5981062. Sample 6 is in accordance with the invention.

Table 3 clearly shows that sample 5, manufactured using the polyamide terpolymer provides higher tenacity retention compared to either of the homopolymers PA6, or PA6,6 used in samples 3 and 4 respectively. In addition, comparison of samples 1, 5 and 6 illustrates the improvement found by combining the polyamide terpolymer and the anti-oxidants in a single blend.

Comparison of samples 1 and 2 shows the advantages of a hindered phenolic based anti-oxidant over a hindered amine, ensuring that this is the preferred primary anti-oxidant. By comparing samples 5 and 6 it is clear that the quantity of polyamide terpolymer used in the blend can be significantly

reduced by utilizing a suitable combination of anti-oxidants. This provides advantages in processing, as the problems described herein associated with blending these two polymers are diminished significantly. This will be further illustrated in a later example.

5 A further embodiment of this invention is the extension of the thermo-oxidative stability of the polyesters described herein such that they are comparable to the industry standard PET yarns. The data indicates that sample 6, which exemplifies this invention retains 80% of its original tenacity after 113 hours at 204°C, whilst PET only retains 66% of its
10 original tenacity following the same test period.

15

20

Example 2

Table 4: Composition of samples 7-12, manufactured following the procedure outlined in Example 2 including extrusion and draw conditions.

	7	8	9	10	11	12
Composition %						
PCTA	98.8	95.8	91.8	91.8	91.85	91.85
Irganox 1010		0.5	0.5	0.5	0.8	0.6
Doverphos S-9228		0.5	0.5	0.5	0.8	
Di-stearyl thiodi-propionate						0.8
Polyamide 6			4			
Polyamide Terpolymer				4	3.35	3.75
Lubricant					0.4	
Color		2	2	2	2	2
Staboxol I	1.2	1.2	1.2	1.2	1.2	1.2
Extruder Profile (F)						
Zone 1	100	100	100	100	100	100
Zone 2	500	500	560	560	560	560
Zone 3	560	560	590	590	570	570
Zone 4	560	580	600	600	580	580
Zone 5	515	515	530	530	530	530
Zone 6	575	575	580	580	580	580
Zone 7	560	580	540	540	540	540
Zone 8	560	560	540	540	540	540
Zone 9	560	560	430	430	530	530
Zone 10	560	560	530	530	530	530
Zone 11	550	550	530	530	530	530
Adaptor	550	550	560	560	560	560
Head temperature setting	558	558	560	560	560	560
Extrusion Conditions						
Die temperature setting	518	518	560	560	560	560
Extruder Pressure (psi)	850	860	1000	1000	1000	1000
Extruder Speed (rpm)	125	125	140	140	125	125
Drawing Conditions						
Total Draw Ratio	3.43	3.43	3.43	3.43	3.43	3.43
Oven 1	300	300	300	300	300	300
Oven 2	320	320	300	300	310	310
Oven 3	330	330	290	290	280	280

Table 5: Physical property results for samples 7-12.

	7	8	9	10	11	12	PET
Tenacity (g/den)	2.6	2.5	2.4	2.6	2.4	2.5	3.8
Elongation @ Break (%)	20	20	20	21	19	20	35
Shrinkage @ 140C (%)			13	12.5	14	13	2
<u>Hydrolysis Resistance Test</u>							
Tenacity Retention After 24 hrs @ 170C Steam (g/den)	0	0	2.16	1.51	2.27	1.62	0
<u>Oven-aging Test</u>							
Tenacity Retention After 89 hrs @204C (g/den)	0	1.7	1.94	1.92	2.16	2.15	2.5
Tenacity Retention After 137 hrs @ 204C (g/den)	0	0	0	1.19	1.94	1.73	1.63

Samples 10, 11 and 12 are in accordance with the invention.

In a further example of the invention, samples of the blends were manufactured using a 147mm co-rotating and intermeshing twin screw extruder. The PCTA resin was dried at 260°F for six hours, the blend components being metered into the polyester using a gravimetric blending system. The composition was fed into the extruder such that the rate of feeding could be controlled. Table 4 provides the compositions and extrusion conditions for the samples. An organic pigment in the form of a masterbatch in a polymeric carrier resin was added to the samples to facilitate observation of filament defects. Table 5 outlines the physical properties, and summarizes the hydrolysis resistance and oven aging tests. Figures 1 and 2 illustrate the performance of the best of these blends relative to PET.

Hydrolysis resistance was measured by placing monofilament samples, wrapped into coils of approximately 5cm diameter, in a pressure vessel or autoclave. The tests were performed at 170°C (7.6atm steam pressure), and 120°C (1.96atm steam pressure). The tenacity retention was measured according to ASTM D2256-97. Oven aging tests were carried out at 175°C and 204°C.

Comparing samples 7 and 8 it can be clearly seen that the antioxidants improve the resistance to oxidation in the oven-aging test. Sample 10, incorporating the polyamide terpolymer, has significantly better oxidative stability than sample 9, that utilizes polyamide 6. The combination of stabilizers used in sample 11 provides the best performance, illustrating that by using a combination of stabilizers, it was possible to reduce the level of the polyamide terpolymer and improve the resistance to the industry standard PET.

Fig. 1 provides a comparison of the hydrolytic stability of sample 12 and PET, tested at 120°C (1atm). Clearly the new composition provides significantly improved property retention over the industry standard PET.

Fig. 2 illustrates the tenacity retention of sample 12 through the oven-aging test at 175°C, with comparison to the industry standard PET. It is evident that this composition has better property retention at elevated temperatures than the industry standard PET.

Example 3

Table 6: Composition of samples 13 and 14, manufactured following the procedure outlined in Example 3 including experimental data.

	13	14
Composition %		
PCTA	90.1	91.85
Irganox 1010	0.2	0.6
Doverphos S-9928	0.2	0.6
Polyamide Homopolymer	4	
Polyamide Terpolymer		3.75
Lubricant	1	
Color	3	2
Staboxol I	1.5	1.2
Extrusion Pressure Range	200	50
Variation (+/-)		
Diameter Variation (%)	1.5	0.7
Filament Anomalies	Present	None

Sample 14 is in accordance with the invention.

In this example, two samples were once again manufactured using a 147mm co-rotating and intermeshing twin screw extruder according to the method outlines in example 2. The compositions, processing data and physical appearance measurements are provided in Table 6. The extruder pressure variation was plotted against time for each of the samples and the data is presented as the maximum pressure range observed during the production of the samples. The diameter was measured using a laser

scanner, commercially available from Lazermike. The data is expressed as the maximum % range of the diameters measured during the production of the filaments. The scan rate, and dwell time on each filament was kept constant.

5 By switching from polyamide homopolymer to a terpolymer, and optimizing amount of anti-oxidants added to the composition, the pressure variation was reduced by a factor of 4. One consequence of this pressure reduction is an improvement in the consistency of the filaments derived. This is clearly illustrated by the improvement in diameter variation when
10 comparing samples 13 and 14.

 The filament anomalies may be defined as short term diameter variation, what are termed 'slubs' by those versed in the art, and other areas of non-uniform structure expressed as short term color variation. Modification of the composition by using less polyamide, and suitable anti-
15 oxidants at optimized addition levels eliminates the anomalies found in filaments extruded from blends of polyamide homopolymer and PCTA.

 It is to be understood that the above described examples are by way of illustration only. Many modifications and variations are possible.

Fig. 1

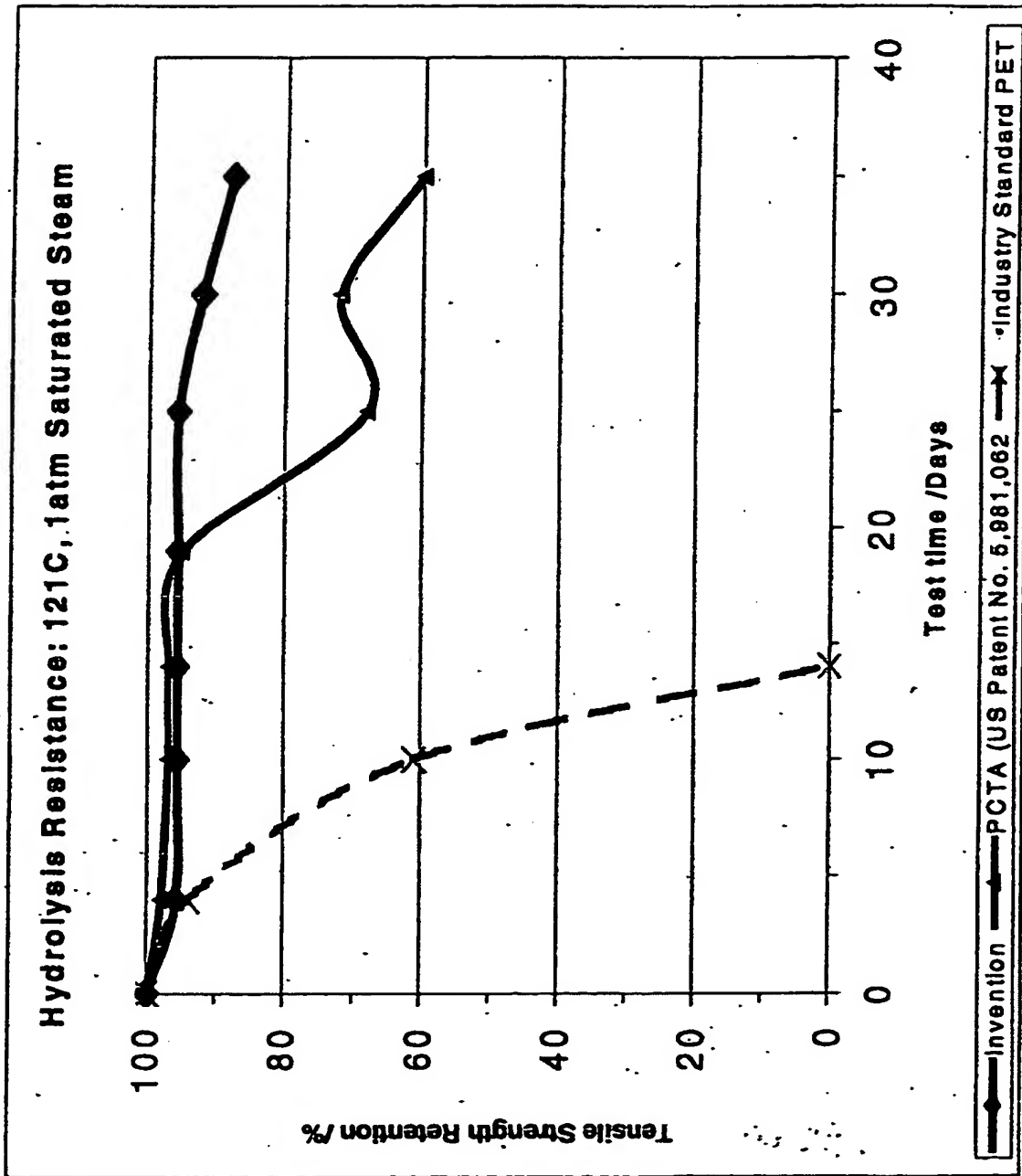
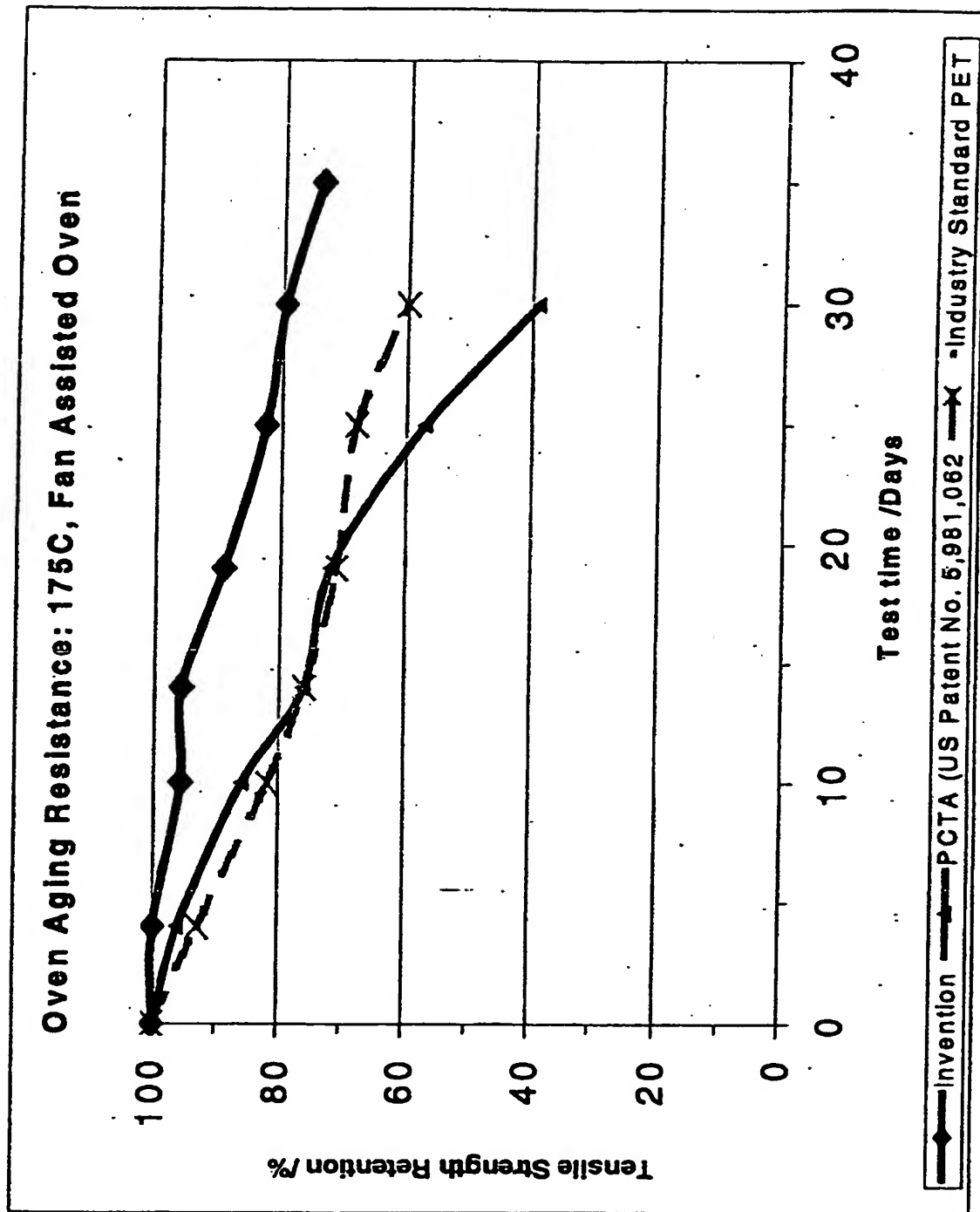


Fig. 2



**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

☒ **BLACK BORDERS**

☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**

☐ **FADED TEXT OR DRAWING**

☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**

☐ **SKEWED/SLANTED IMAGES**

☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**

☐ **GRAY SCALE DOCUMENTS**

☒ **LINES OR MARKS ON ORIGINAL DOCUMENT**

☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**

☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.